



# Promoted-Ignition Testing to Determine the Gaseous Oxygen Compatibility of the Intermetallic Compound 60-NITINOL

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## Summary

The flammability of 60-NITINOL (60 wt%Ni-40 wt%Ti) in an oxygen-rich atmosphere is assessed. It is determined that 60-NITINOL burns readily in gaseous oxygen and would not be a good candidate for components exposed to oxygen-rich environments where there may be an ignition source. The results are the same whether the material is tested without heat treatment, after a solution treatment or after furnace annealing. These results provide guidance for materials selection of aerospace turbomachinery components.

## Introduction

60-NITINOL (60 wt%Ni-40 wt%Ti) is an intermetallic compound that has been found to have a unique combination of high hardness, low apparent elastic modulus, moderate density, high resistance to aqueous corrosion and excellent tribological properties (Refs. 1 to 3). This material has already been identified as an outstanding candidate for aerospace applications like gears and bearings and additional applications are being explored.

An inevitable question to be addressed is whether or not this material could be used in rocket engine propulsion turbomachinery systems (e.g., rocket engine turbopumps and compressors). The initial screening test for materials under consideration for this type of application is an evaluation of the flammability of the material in an oxygen-rich environment. Previous studies have shown that Ni-rich alloys have superior compatibility with gaseous oxygen but an increasing concentration of a second, easily-ignitable metal (such as Ti) reduces the ignition resistance of binary alloys (Refs. 4 to 4). For example, Table I shows that, for increasing concentrations of Ti in Ni-Ti, the *threshold pressure* (the minimum pressure required for sustained ignition of the material in a promoted-ignition test) tends to decrease. One study found that 56 wt%Ni-44 wt%Ti (close in composition to 60-NITINOL and, thus, possibly similar in its gaseous oxygen compatibility) was flammable in gaseous oxygen at pressures of 0.14 MPa and greater. This increase in flammability compared to that of compounds with lower concentrations of

Ti was attributed to the presence of a eutectic (that actually does not exist at this composition) and an intermetallic, which, based on the phase diagram shown in Figure 1, one can only assume is  $\gamma$ -NiTi (hereafter referred to as NiTi) (Ref. 6). If the authors, on the other hand, refer to the intermetallic  $\beta$ -Ni<sub>3</sub>Ti (hereafter referred to as Ni<sub>3</sub>Ti), which they discuss earlier in the same paragraph of their paper, it is unclear from the phase diagram how this compound would form since 56 wt%Ni-44 wt%Ti would only form NiTi when cooled from above approximately 850 °C or higher. However, the heat treatment history of these specimens is not given. Therefore, further investigation was required to determine the flammability of 60-NITINOL in oxygen-enriched environments. The purpose of this investigation was to determine the compatibility of 60-NITINOL in gaseous oxygen for specimens that have well-characterized heat treatment histories.

## Materials and Procedures

Three rods were sectioned from cast 60-NITINOL (60wt%Ni-40wt%Ti). The rod dimensions were 3.2 mm diameter by 152 mm long. One rod was not heat treated (designated: *as-cast*). Another rod was treated at 1050 °C for 2 h in argon then quenched in room temperature water. The third rod was heat treated for 2 h at 1050 °C after which the furnace was shut off, left closed and allowed to cool to room temperature. These heat treatment processes are referred to as *solution treating* and *furnace annealing* in this study. Solution treating is designed to dissolve the second phase Ni<sub>3</sub>Ti resulting in austenitic NiTi. This corresponds to the condition 60-NITINOL would be given prior to service as it results in hardness typically greater than 60 HRC. Furnace annealing allows more of the second phase Ni<sub>3</sub>Ti to precipitate resulting in a softer material (approximately 33 HRC) with minimal residual stresses. To minimize surface oxidation, each heat treatment was done in an argon atmosphere. The heat treated specimens were also ground lightly with abrasive paper to remove the trace oxide layer. Representative specimens were prepared for metallographic examination and tested for hardness.

TABLE I.—THRESHOLD PRESSURES FOR SEVERAL BINARY Ni-Ti COMPOUNDS (REF. 4)

Wt% Ti in Ni-Ti	Threshold pressure, MPa
1.0	>69
3.2	>69
5.0	>69
8.3	31.0
26	0.14
44	0.14

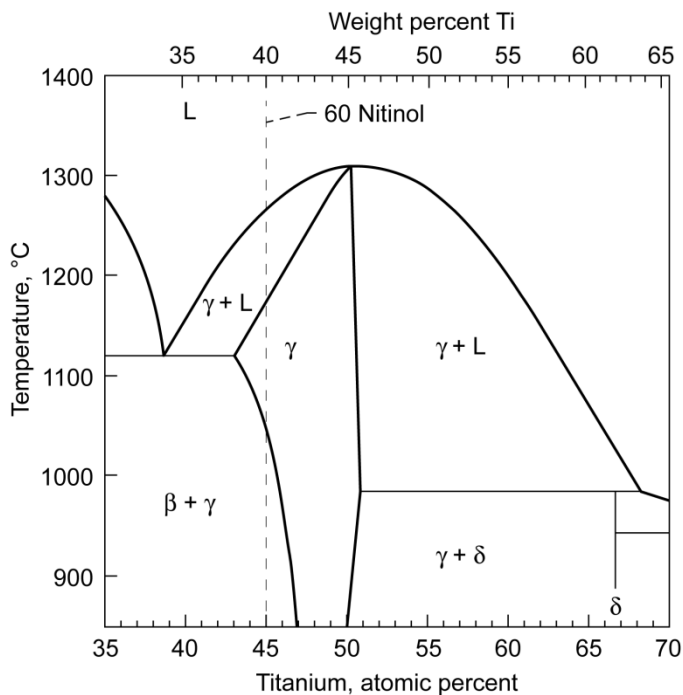


Figure 1.—A portion of the Ni-Ti phase diagram above 850 °C (Ref. 6).

Each rod was tested for gaseous oxygen compatibility according to standard test methods (Refs. 7 to 8). A schematic of the experimental setup is shown in Figure 2. For each test, an aluminum or magnesium promoter was affixed to the end of the specimen. A small diameter wire composed of aluminum enclosing a palladium core was wrapped around the promoter and connected to a power supply. The test sample was loaded into the test sample holder and the gaseous oxygen pressure in the test chamber was increased to 0.14 MPa at room temperature. The atmosphere was selected to match the threshold pressure at which the 56wt%Ni-44wt%Ti compound burned in the previously cited study (Ref. 4). Sufficient

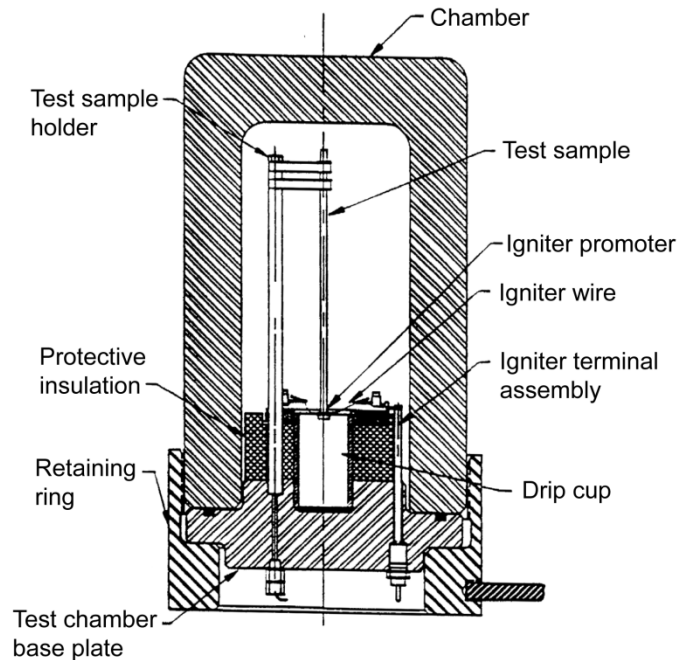


Figure 2.—Schematic of typical test setup for promoted-ignition test of gaseous oxygen compatibility (Ref. 7).

current was supplied to provide approximately 3 kJ of heat to the igniter wire and ignite the promoter. Once the promoter was ignited, the power supply was turned off and the length the specimen burns was recorded. The molten slag from each promoted-ignition test was collected in a copper drip cup filled with a ceramic material for adsorption.

## Results and Discussion

Optical photomicrographs showing the typical microstructures of as-cast, solution treated and furnace annealed 60-NITINOL prior to testing are shown in Figure 3. A filter is used to provide monochromatic (green) light for increased image resolution and the original magnification of each image is 200×. The furnace annealed and as-cast microstructures have more precipitated  $\text{Ni}_3\text{Ti}$  along the grain boundaries and in a lath-type pattern formed from layers of  $\text{Ni}_3\text{Ti}$  within the grains. The solution treated material has traces of residual  $\text{Ni}_3\text{Ti}$  near the grain boundaries of the material. The parent phase in each of the photomicrographs is NiTi. The Rockwell hardness of the material from each heat treatment condition is given in Table II. The average and standard deviation of three to five duplicate hardness measurements per specimen is reported.

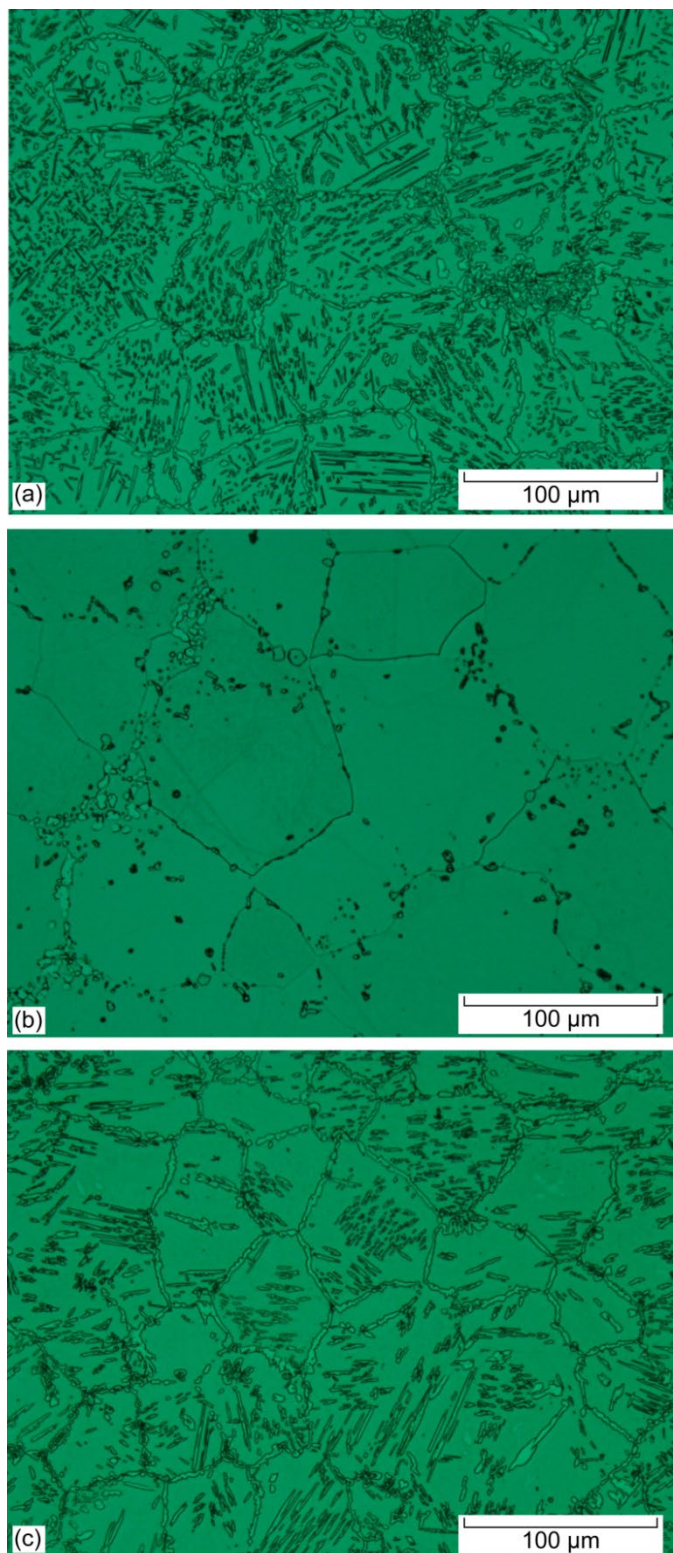


Figure 3.—Microstructure of 60-NITINOL (a) as-cast, (b) after solution treating (2 h at 1050 °C followed by a water quench) and (c) after furnace annealing (2 h at 1050 °C followed by furnace cooling).

TABLE II.—HEAT TREATMENTS AND RESULTANT HARDNESS OF CAST 60-NITINOL

Designation	Heat treatment	Hardness, HRC
As-cast	None	31 ± 3
Solution treated	2 h at 1050 °C/ water quenched	63 ± 1
Furnace annealed	2 h at 1050 °C/ furnace cooled	33 ± 2

The 60-NITINOL specimens burned readily at 0.14 MPa in gaseous oxygen in each of the three heat treatment conditions considered in this study. The entire length of each specimen was consumed after ignition. This result corroborates previously reported findings (Ref. 4) and, furthermore, provides a more complete description of the thermal histories and microstructural conditions of the specimens prior to testing. While Tayal et al. (Ref. 4) claim that the binary Ni-Ti compound containing 43.9 wt%Ti (that burned at 0.14 MPa) was a combination of an intermetallic and a eutectic compound, the results reported herein confirm that intermetallic NiTi, with or without substantial amounts of intermetallic Ni<sub>3</sub>Ti, burns readily at 0.14 MPa. It is possible that 60-NITINOL could be used in *hydrogen-rich* environments (e.g., hydrogen pumps and valves). However, additional work would be required to determine the compatibility of 60-NITINOL with hydrogen-rich atmospheres and to address the potential issue of hydrogen embrittlement.

The slag from each test was cross sectioned and analyzed metallographically. Figure 4 shows optical photomicrographs of the slag from each specimen. Each image shows a dendritic structure indicating the prior molten state of the material. Backscattered electron image scanning electron photomicrographs of the as-cast specimen slag (shown in Figure 5) indicates compositional differences within the specimen. X-ray spectra of the different regions on this specimen (shown in Figure 6) indicate the presence of Ni, Si, Pd, and a trace amount of Fe. The promoter wire is the most likely source of the Pd. The adsorption material in the drip cup (a silicate compound) is the most likely source of the Si. The reason for the absence of Ti, Mg and Al from the x-ray spectra is unknown. Since these are all lighter elements, it is possible that they oxidized during the test and drifted away rather than being collected in the drip cup.



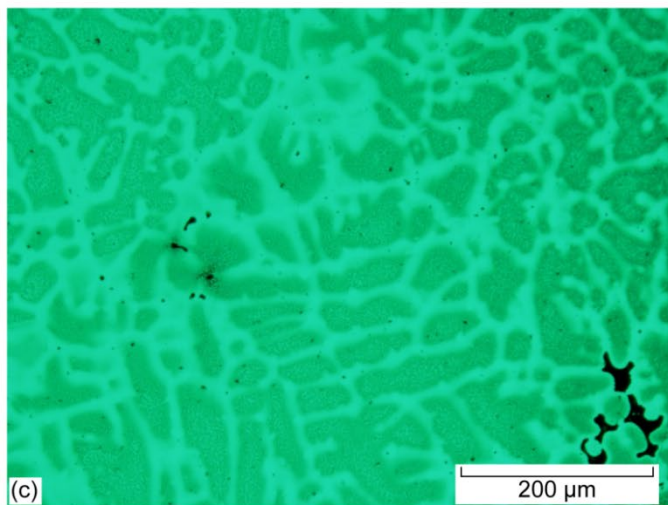
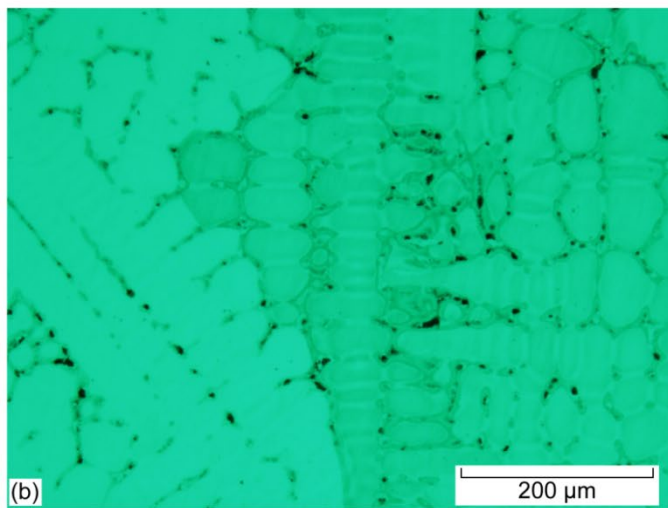
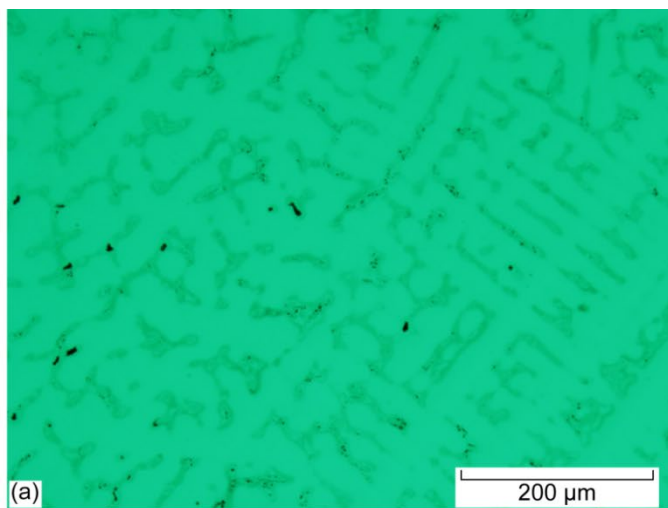


Figure 4.—Optical photomicrographs of slag collected from (a) the as-cast specimen, (b) the solution treated specimen and (c) the furnace annealed specimen (100× original magnification).

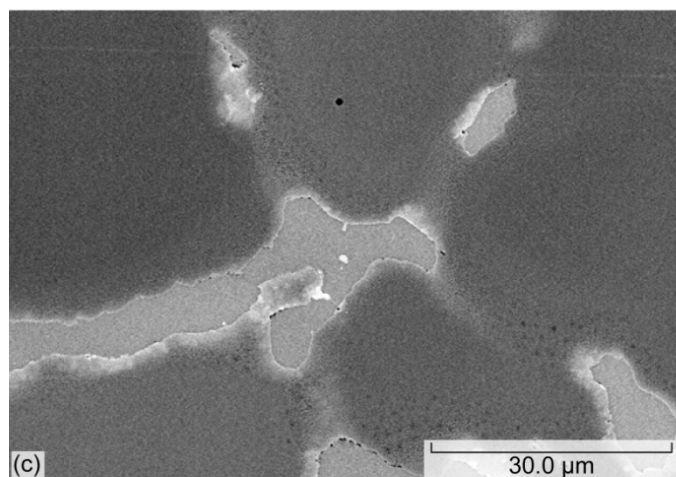
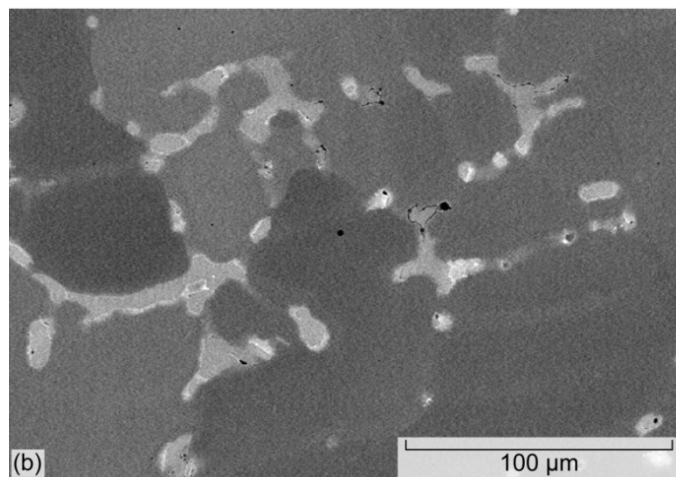
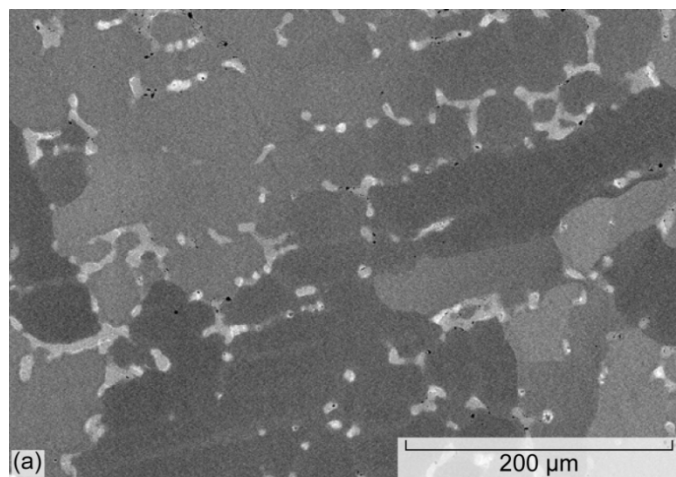


Figure 5.—Scanning electron photomicrographs of slag from as-cast specimen shown in Figure 4(a) at (a) 250×, (b) 500× and 1500× original magnification.



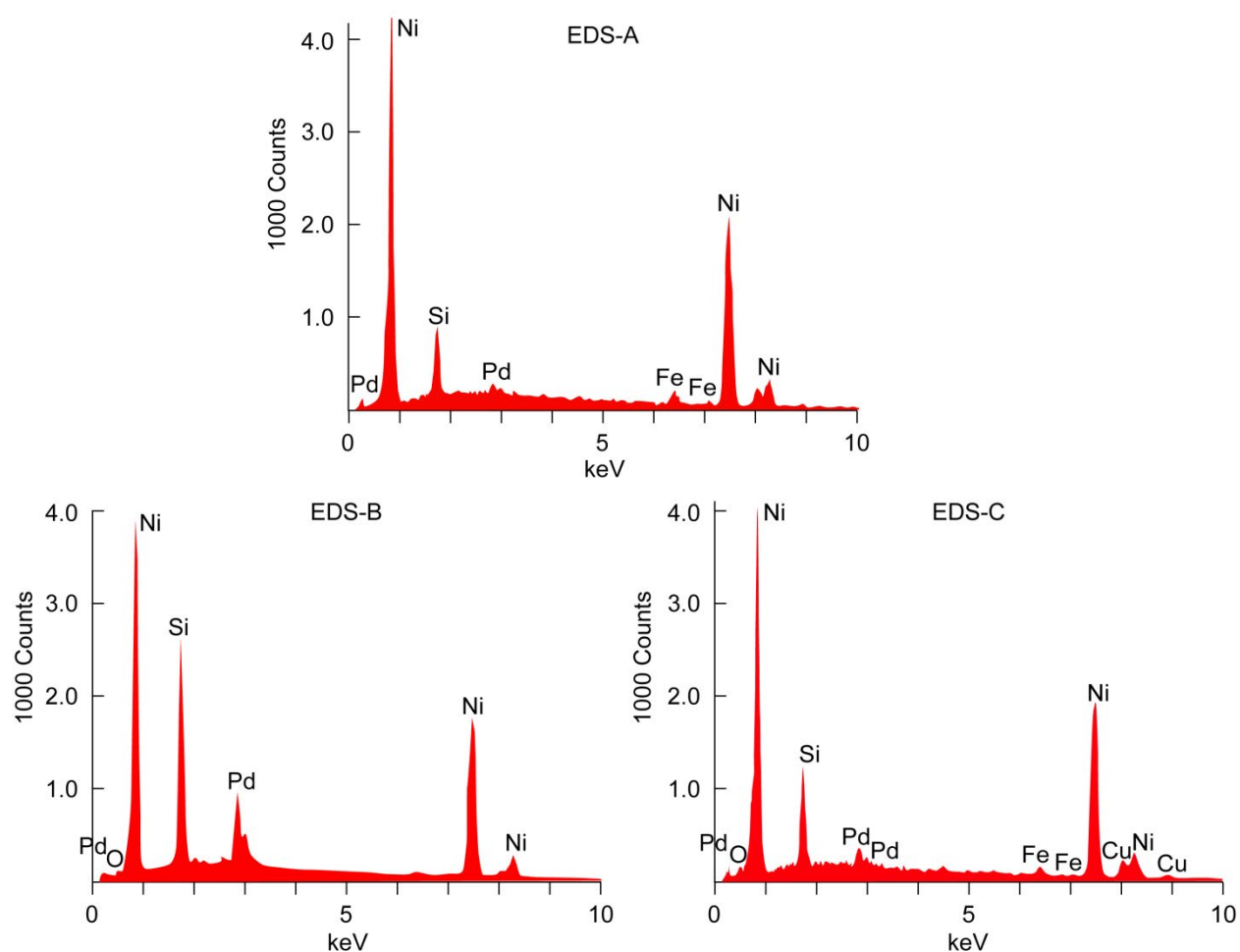
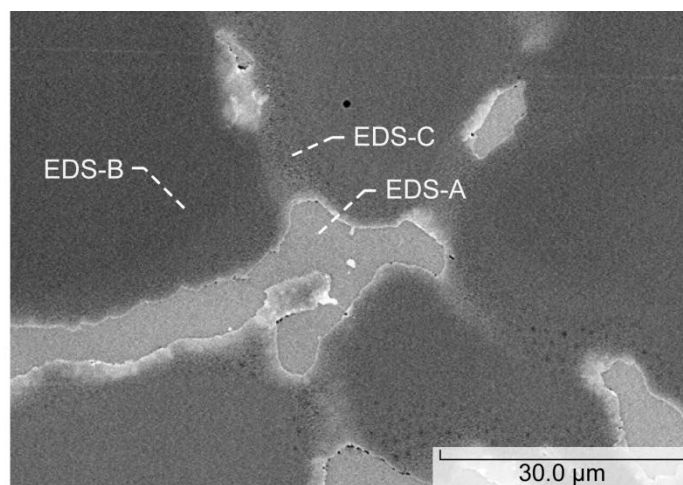


Figure 6.—Scanning electron photomicrograph from Figure 5(c) and energy-dispersive x-ray spectra from slag collected from as-cast 60-NITINOL specimen after promoted-ignition test.

## Concluding Remarks

The promoted-ignition performance of 60-NITINOL has been evaluated. Since 60-NITINOL has been found to sustain ignition at 0.14 MPa, it can be concluded that this material would not be a good candidate material for mechanical components operating in oxygen-rich environments. This statement is especially applicable when there may be an unexpected transient ignition source such as a spark or an asperity flash from highly localized sliding friction. These results may also provide guidance to other fields considering the use of 60-NITINOL where flammability of materials poses a hazard such as oxygen production and the diving equipment industry.

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